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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF : Tomoyuki Maeda et al.
SERIAL NUMBER : 10/050,118
FOR : MAGNETIC RECORDING MEDIUM
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EXAMINER : Bernatz, Kevin M

DECLARATION UNDER 37 C.F.R. 1.132

Assistant Commissioner for patents
Washington, D.C. 20231

Sir:

I, Akira Kikitsu, a national of Japan, declare as follows.

I am a co-applicant of the above-identified application.

The following Experiments were conducted by me or under my direct supervision:

Experiments

(1) Manufacture of magnetic recording media in accordance with the method discussed in Coffey et al.

Two magnetic recording media, each having an Fe/Pt/Au magnetic layer whose composition was 55/34/11 or 50/38/12, as described in Table in Column 12 of Coffey et al., were prepared.

Coffey et al. does not provide a detailed description on the manufacturing method, and therefore the sputtering, for example, was carried out under standard conditions. As described in Column 11, line 14, a silicon disk substrate with a surface layer of native oxide was used in each sample. In order to adjust the composition of the magnetic layer, ternary simultaneous sputtering was carried out with use of three targets of Fe, Pt and Au. As the sputtering gas, Ar (having a purity of 5N (five nine)), which is ordinarily used, was used. The sputtering pressure was set to 1Pa, which is an ordinarily condition in the manufacture of magnetic recording media. An FePtAu layer having a thickness of 10nm was formed while controlling the composition of the layer by adjusting the deposition rate, which is achieved by varying the power applied to each target. Then, a carbon protective layer was formed by sputtering in an Ar

atmosphere to which 10% of hydrogen by flow ratio was added.

The composition of the magnetic film was analyzed by Inductive Coupled Plasma Spectroscopy (ICP). The results of the composition analysis of the magnetic layers were as follows:

sample A: $\text{Fe}_{55}\text{Pt}_{34}\text{Au}_{11}$ i.e., $(\text{Fe}_{0.62}\text{Pt}_{0.38})_{89}\text{Au}_{11}$

sample B: $\text{Fe}_{50}\text{Pt}_{38}\text{Au}_{12}$ i.e., $(\text{Fe}_{0.57}\text{Pt}_{0.43})_{88}\text{Au}_{12}$

Each magnetic layer thus formed was annealed under flowing $\text{Ar}/4\%\text{H}_2$ gas in an atmospheric pressure by a quartz lamp furnace in accordance with the description provided in the reference, column 8, lines 32 to 34. The annealing conditions were 500°C for 10 minutes and 300°C for 10 minutes.

The coercivity of each sample after the annealing was as follows:

	500°C	300°C
sample A:	6.2kOe	0.2kOe
sample B:	7.1kOe	0.2kOe

These values are somewhat different from those described in Coffey et al.; however, these differences are due to the use of a different sputtering apparatus. It was judged that the differences were not significant and these samples were equivalent to those discussed in Coffey et al.

Next, the fine structure of each sample was examined by a plan view TEM image. The crystal grain size was as follows: about 35nm in the case of annealing at 500°C and about 10nm in the case of annealing at 300°C for sample A, whereas about 40nm in the case annealing at 500°C and about 10nm in the case of annealing at 300°C for sample B. These values are also different from those described in Coffey et al. due to the use of a different sputtering apparatus. Between crystal grains, there were grain boundary regions that are deduced to be made of Fe-O compound. Cross sectional TEM observation revealed that there was an amorphous initial layer (dead layer) having a thickness of about 3nm between the substrate and the FePtAu layer. Thus, it was deduced that an oxide was present on the substrate.

A region of the FePtAu layer which does not include the initial layer, for

example, a region 2nm-deep from the surface of the film was selected, and the composition in the crystal grains was analyzed. The analysis was performed with TEM-EDX having a probe diameter of 2nm. The results of the analysis were as follows:

Annealed at 500°C

sample A: $\text{Fe}_{30}\text{Pt}_{53}\text{Au}_{17}$ i.e., $(\text{Fe}_{0.36}\text{Pt}_{0.64})_{83}\text{Au}_{17}$

sample B: $\text{Fe}_{28}\text{Pt}_{55}\text{Au}_{17}$ i.e., $(\text{Fe}_{0.34}\text{Pt}_{0.66})_{83}\text{Au}_{17}$

Annealed at 300°C

sample A: $\text{Fe}_{33}\text{Pt}_{51}\text{Au}_{16}$ i.e., $(\text{Fe}_{0.39}\text{Pt}_{0.61})_{84}\text{Au}_{16}$

sample B: $\text{Fe}_{32}\text{Pt}_{52}\text{Au}_{16}$ i.e., $(\text{Fe}_{0.38}\text{Pt}_{0.62})_{84}\text{Au}_{16}$

In each sample, the Fe content of the crystal grain was 40at.% or less and the Pt content was 60at.% or higher, which were out of the range defined in the present invention.

Less Fe content shown above might be attributed to the formation of the initial layer and the grain boundary regions. The width of the grain boundary regions was as large as 1 to 2nm even in a narrower part. This means a significantly high volume fraction of boundary portion. The thickness of the initial layer was about 30% with respect to the total thickness of the magnetic layer. The composition of the initial layer portion was analyzed with cross section TEM. As a result, it was found that these portions were made of mostly Fe-O. Further, qualitative composition analysis was performed by means of XPS, and it was confirmed that a large amount of Fe was oxidized into Fe-O. Therefore, oxygen from the sputtering gas and substrate oxidized Fe element in the FePtAu samples..

(2) Manufacture of magnetic recording media in accordance with the method described in the present invention:

Two magnetic recording media, each having an Fe/Pt/Au magnetic film whose composition was 55/34/11 or 50/38/12 (same as (1) described above), were prepared in accordance with the method described in the specification of the present invention, page 19, line 25 to page 20, line 16.

An MgO target was RF-sputtered in Ar to form an MgO underlayer

having a thickness of 10nm on a substrate, and FePtAu magnetic layer and a protective layer were subsequently deposited without breaking vacuum. The composition of the magnetic layer was analyzed by ICP. The results of the composition analysis of the magnetic layers were as follows:

sample A2: $\text{Fe}_{55}\text{Pt}_{34}\text{Au}_{11}$ i.e., $(\text{Fe}_{0.62}\text{Pt}_{0.38})_{89}\text{Au}_{11}$

sample B2: $\text{Fe}_{50}\text{Pt}_{38}\text{Au}_{12}$ i.e., $(\text{Fe}_{0.57}\text{Pt}_{0.43})_{88}\text{Au}_{12}$

The magnetic layers thus formed were annealed in a pure hydrogen atmosphere in such a manner as described in the specification of the present invention, page 20, lines 13 to 16. The annealing condition was set to 500°C for 10 minutes or 300°C for 10 minutes.

The coercivity of each sample after the annealing was as follows:

	500°C	300°C
sample A2:	9.2kOe	6.5kOe
sample B2:	8.3kOe	5.7kOe

Unlike the case of Coffey et al., a high coercivity was obtained in every case. This is because the ordering process progressed even under an annealing at 300°C.

Next, the fine structure of each sample was examined by a plan view TEM image. The crystal grain size was as follows: about 50nm in the case of annealing at 500°C and about 35nm in the case of annealing at 300°C for sample A2, whereas about 60nm in the case of annealing at 500°C and about 35nm in the case of annealing at 300°C for Sample B2. The crystal grain size is larger in each case as compared to the case of Coffey et al. The crystal grains were identified easily by the contrast from the variation in the crystal orientation. However, the grain boundary region, which corresponds to the Fe-O grain boundary regions between crystal grains observed in (1) above, was not clear. Cross sectional TEM observations showed a thin initial layer having a thickness of about 1nm between the substrate and FePtAu film, although it was not clear.

A region of the FePtAu layer, which does not include the initial layer, for

example, a region 2nm-deep from the surface of the film was selected, and the composition in the crystal grains was analyzed. The analysis was performed with TEM-EDX having a probe diameter of 2nm. The results of the analysis were as follows:

Annealed at 500°C:

sample A2: $\text{Fe}_{49}\text{Pt}_{38}\text{Au}_{12}$ i.e., $(\text{Fe}_{0.56}\text{Pt}_{0.44})_{88}\text{Au}_{12}$

sample B2: $\text{Fe}_{46}\text{Pt}_{41}\text{Au}_{13}$ i.e., $(\text{Fe}_{0.53}\text{Pt}_{0.47})_{87}\text{Au}_{13}$

Annealed at 300°C:

sample A2: $\text{Fe}_{51}\text{Pt}_{37}\text{Au}_{12}$ i.e., $(\text{Fe}_{0.58}\text{Pt}_{0.42})_{88}\text{Au}_{12}$

sample B2: $\text{Fe}_{44}\text{Pt}_{42}\text{Au}_{13}$ i.e., $(\text{Fe}_{0.51}\text{Pt}_{0.49})_{87}\text{Au}_{13}$

As indicated above, in any of the samples that were annealed at 500°C or 300°C, the Fe content and Pt content of the crystal grain fell within the range specified in Claim 1 of the present invention.

The difference between the experiment results of (2) and those of (1) is attributed to the difference in the amount of Fe-O, as is clear from the comparison between the results of the composition analysis by means of plan view TEM and cross section TEM described above.

One of the reasons why the difference in the amount of Fe-O results is the presence or absence of an underlayer (MgO). In general, the underlayer blocks diffusion of oxygen from the substrate. Further, an MgO layer formed by the sputtering in Ar, as in the experiment (2), generally has less oxygen than stoichiometric composition. Therefore, the MgO underlayer absorbs oxygen supplied as an impurity of the Ar gas during the sputtering, which prevents oxygen from being contained in the FePtAu layer.

Another reason of the difference in the amount of Fe-O is due to the difference in annealing atmosphere. The Ar/4%H₂ gas employed in the experiment (1) provides a reducing atmosphere, but it is still insufficient to eliminate oxygen from the FePtAu layer, and further it is not possible to avoid diffusion of oxygen, which is contained as an impurity in the Ar atmosphere, into the FePtAu layer. In comparison, the pure hydrogen atmosphere employed in

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the experiment (2) is effective to eliminate oxygen from the FePtAu layer.

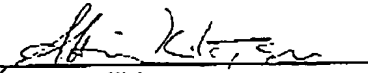
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I, the undersigned, declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Date

Aug. 4, 2003


Akira Kikitsu